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Research Article

Preparation of Briquette by Brown Coal Gangue-blast Furnace Slag Geopolymer Binder and Its Characteristics

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Abstract

Effective recycling of industrial waste is a very important issue worldwide. Coal gangue is a solid waste generated during coal production and processing, which accounts for 10-20% of coal production, the largest of industrial wastes discharged so far. Geopolymers are three-dimensional amorphous inorganic polymers in the form of Si-O-Al-O-Al, in which silica and alumina precursors with high reaction activity are formed via depolymerization-condensation processes in a highly alkaline environment. In this paper, a geopolymer binder is prepared by combining an alkaline activator prepared from brown coal gangue and blast furnace slag as raw material and from industrial waste silica fume. Also, the properties of these geopolymer binders are examined using them as a briquette binder. At temperatures above 700 ℃, roasted brown coal gangue is more active than the initial state. The optimum dosage of alkali activator is 10M NaOH, silica fume/NaOH ratio of 3, specific gravity of 1.42, and the addition of binder of 6%. The main polymerization products of the alkali activated brown coal gangue geopolymer samples are N-A-S-H gel and amorphous aluminosilicate gel, while the main polymerization products of the alkali activated brown coal gangue -blast furnace slag geopolymer samples are N-A-S-H gel, C-(A)-S-H gel and amorphous aluminosilicate gel. Blast furnace slag is added during the preparation of briquette binder by brown coal gangue geopolymer, which increase the mechanical strength of the geopolymer binder and the optimum dosage is 30%.

Keywords

Geopolymer, Briquette, Binder, Coal Gangue, Aluminosilicate

1. Introduction

Geopolymer, also called "geopolymer cements," are formed by the destruction of Si-O and Al-O bonds in the raw material in the presence of alkalis, forming AlO_4 and SiO_4 tetrahedral structural elements. Then, the polycondensation leads to the formation of an inorganic polymer with a network of O-Si-O-Al-O. As a silicon aluminum source material, the main reaction materials are sheet kaolin and fly ash, and alkali activator is used. Generally, the geopolymers produced by the reaction of calcium-containing aluminosilicate materials with alkaline materials are called alkali-activated materials [1, 2]. Many methods have been reported for the preparation of low-calcium or calcium-free aluminosilicate materials (e.g., kaolinite, coal fly ash, coal gangue, etc.) and polymers prepared

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by reaction with alkali activators [3, 4].

There are many studies using coal gangue as geopolymer raw material. The geopolymer is prepared from roasted coal gangue, granulated blast furnace slag and slag, and low calcium content in coal gangue was found to be the main reason for the low strength of geopolymer [5]. In another study, 20% of coal gangue was added to the sludge to produce high strength slurry-coal gangue geopolymer, but the optimum application index values were not obtained [6]. Sludge- coal gangue composite polymer is prepared and the alkaline chemical components K_2O and Na_2O in the sludge are reported to enhance the compressive strength by undergoing polymerization reaction with the burned coal gangue [7].

In another study, NaOH, KOH and water glass are reacted with coal gangue, and the formation of an amorphous alkaline aluminosilicate gel similar to the zeolite precursor structure is observed by SEM [8]. Coal gangue-slag coagulation materials are prepared using water glass as activator, and the compressive strength of geopolymer are observed to be higher than 40MPa when the amount of coal gangue was less than 30% [9]. The early hydration of coal gangue with $Ca(OH)_2$+water glass and $Ca(OH)_2$+NaOH as complex alkali activators, respectively, is studied and found that the hydration process of coal gangue + $Ca(OH)_2$ + water glass system is more stable, with the presence of inductive groups and more compact internal structure [10]. Studies have confirmed that coal gangue can be a raw material for the production of geopolymers. The briquette binder is the key technology of briquettes, is also an important guarantee affecting the final quality of briquettes, and has been a major factor limiting briquette development for a long time [11]. The type of briquette binder is very large and can be divided into three categories: organic, inorganic and composite binders in a broad sense. Organic binders can penetrate coal material well, even penetrate the pore structure of coal, and can be hard-bonded to coal material after dry curing [12]. Therefore, the bonding performance of organic binder is relatively good and has relatively high mechanical strength after briquette drying, but the strength after heat treatment is not high because the organic matter is easily decomposed and combusted at high temperature. Since the characteristics of inorganic binders are stable in performance and mostly unburnt, the addition of inorganic binders can reduce the calorific value of the briquettes by increasing briquette ash [13, 14]. However, the source of inorganic binders is abundant, relatively cheap, and has a higher strength after heat treatment compared to organic binders. Composite binders are preferred in a manner that takes advantage of organic and inorganic binders and adds defects, and are therefore currently the main research direction of briquette binders.

In this paper, the briquette binder based on geopolymer prepared from industrial waste is prepared and its properties are examined.

2. Materials and Method

2.1. Raw Materials

The chemical composition of the brown coal gangue used in the experiment is shown in Table 1. The purity of NaOH is 99.5%.

Table 1. *Main chemical composition of raw materials/wt%.*

chemical composition	brown coal gangue	blast furnace slag	Silica fume
SiO_2	64.8	37.5	73.7
Al_2O_3	29.8	13.1	1.3
Fe_2O_3	3.5	2.5	0.9
CaO	0.7	39.7	7.0
MgO	1.1	1.6	2.0

2.2. Experimental Method

2.2.1. Preparation of Alkali Activated Solutions

Add 100g of water to 42g of NaOH and stir for 30min with a magnetic stirrer to make an aqueous solution of NaOH. At a certain dosage, silica fume is added to the above prepared aqueous NaOH solution, stir thoroughly for 1h with a magnetic stirrer and incubated for 24h at room temperature.

2.2.2 Preparation of Geopolymer Briquette Binder

After activation by roasting the brown coal gangue at 700℃ for 2h, it was mixed with the crushed blast furnace slag at different proportions. Then, the above-prepared alkaline activation solution was put into the reactor at a ratio of 6:4 and prepared as a pellet binder by geopolymerization with mechanical stirring.

2.2.3. Briquette Moulding

The prepared binder is mixed and blended with a certain proportion of the crushed anthracite to a certain size and then molded, at that time, molding moisture to be about 10~12%. Molded briquettes are dried in a drying furnace at 100~120℃ for 2 hours to ensure moisture content of less than 0.5%.

2.2.4. Structural Analysis of Geopolymer Samples and Measurement of Mechanical Strength of Briquettes

(1) Compressive strength measurements

The compressive strength of the briquette is measured using a hydraulic compressive strength measuring device (ZCES-300). After heat treatment, the strength is measured by cooling

them to room temperature after 30min of storage at 850℃ in a sand-covered atmosphere using a mortar.

(2) XRD analysis

D8-ADVANCEX-type X-ray powder diffractometer is used. The Cu-Kα target radiation, scan rate of 2°/min, step length of 0.02 °, and sample shall be scanned continuously at 5° to 70° (2θ).

(3) FT-IR analysis

The functional group of the sample is evaluated by a Nicolet 6700 infrared spectrometer, with a range of 4,000cm-1 to 400cm-1 measurements, a resolution of 0.09cm-1, and a dynamic adjustment rate of 1300,000rpm.

(4) SEM analysis (data entry)

The microstructure of geopolymer is observed using a SU8010-type field emission scanning electron microscope, with an electron beam accelerating voltage of 15kV.

(5) TG-DTG analysis

Thermogravimetric-differential thermal analysis tests are performed using a DERIVATOGRAPHQ-1500D-type thermogravimetric analyzer, with the temperature rise from room temperature to 600 ℃ within the test time and the temperature rise rate was controlled at 10 ℃/min in a nitrogen gas protection environment.

3. Results and Discussion

3.1. Effect of Roasting of Brown Coal Gangue on Activity

Figure 1 and Figure 2 show XRD and SEM images of the roasted and unburned brown coal gangue. As shown in Figure 1, the diffraction peak of kaolinite in brown coal gangue decreased after roasting at 700 ℃, and the diffraction peak of quartz enhanced. From Figure 2, it can be seen that the structure of the unburned brown coal gangue is very dense and has a relatively stable crystalline structure. However, the microstructure of brown coal gangue calcined at 700℃ was destroyed, the structure was very loose.

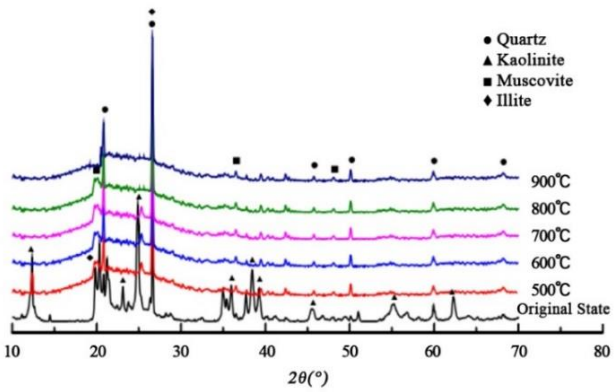
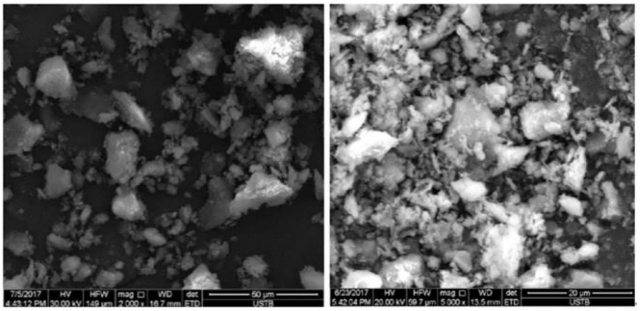


Figure 1. *XRD of Original State of Brown coal gangue and at different temperatures roasted brown coal gangue.*



(a) Original state (b) brown coal gangue roasted at 700℃

Figure 2. *SEM micrographs of the original and roasted cases of brown coal gangue.*

3.2. Effect of Varying Alkaline Activation Solution on the Properties of Geopolymer

Figure 3 shows the compressive strength results of the specimens with different NaOH molar concentrations, with a fixed silica fume /NaOH mass ratio of 2.5. When the NaOH molar concentration increased from 6 to 14M, the compressive strength values of the specimens increased and compared to the 28day compressive strength of the 6M specimens, the compressive strength of the 8M, 10M, 12M and 14M specimens showed an increasing trend, while the strength increase was more pronounced when the NaOH molar concentration increased from 8 to 10M and 12M. At 10, 12 and 14 M molar concentrations of NaOH, the compressive strength values after 28 days of the specimens did not increase significantly, and from a cost point of view, the molar concentrations of NaOH were found to be optimal at 10 and 12M. The compressive strength of the specimens is very low when the mole concentration of NaOH is less than 6M. This is due to the low alkali concentration that prevents the geopolymerization reaction from proceeding properly.

The compressive strength of the briquette with geopolymer binder increased with increasing molar concentration of NaOH, which could be attributed to the higher molar concentration of NaOH, which facilitated the geopolymerization process by dissociating more active SiO_2 and Al_2O_3. On the other hand, when the NaOH molar concentration is higher than 14M, the high concentration of NaOH will cause the initial precipitation of aluminosilicate gel, hindering the polycondensation of SiO_4 and AlO_4, which will have a negative effect on the mechanical strength. It can be seen that the strength after 3days of testing is much higher than that of concrete specimens with normal cement, but the later strength is lower. This may be due to the low CaO content in the brown coal gangue which promotes the hydration reaction.

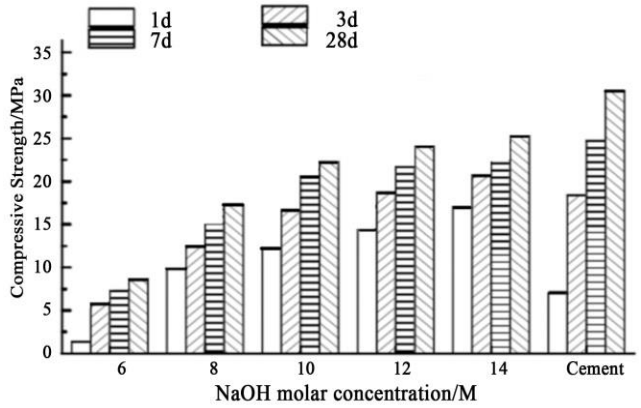


Figure 3. *Compressive strength of brown coal gangue waste geopolymer specimens with varying NaOH molar concentration.*

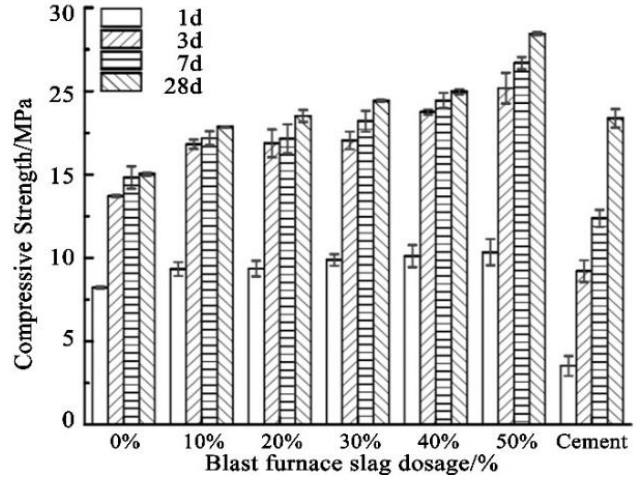


Figure 4. *Compressive strength of geopolymer specimens with the addition of blast furnace slag.*

Figure 4 shows the results of the compressive strength of the specimens with varying amounts of blast furnace slag. The addition of blast furnace slag resulte in a much higher initial compressive strength compared to the non-added brown coal gangue geopolymer samples. The compressive strength of the geopolymer specimens make by adding appropriate amount of blast furnace slag to brown coal gangue was higher than that of the Portland cement specimens after 28d, as well as the compressive strength after 3d.

3.3. XRD Analysis Results of Brown Coal Gangue-blast Furnace Slag Geopolymer Binder

Figure 5 shows the X-ray diffraction patterns of the samples with different slag dosages, the main characteristic peaks are SiO_2, $CaAl_5Si_{20}O_{10}$ $4H_2O$, $NaAlSiO_2$ H_2O, $AlSi_2O_5$ xH_2O, and the main crystalline components are quartz, calcite and zeolite (aluminosilicate crystals). It can be seen from the Figure that the gelling material contains many inert quartz that does not participate in chemical reactions, i.e. this geopolymer binder is a mixture of reactants and unreacted products.

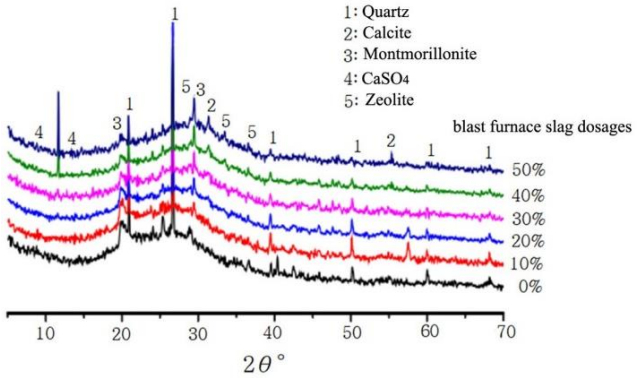


Figure 5. *XRD of geopolymer binder with different blast furnace slag dosage.*

The XRD spectra show that the presence of a calcium zeolite (Ca $[Al_2Si_3O_{10}]$ $3H_2O$) diffraction peak at 30° to 40° of 2θ° was observed, and the formation of a C-A-S-H gel phase is observed. Next, with increasing slag dosage, the diffraction peak of SiO_2 decreases and the diffraction peak of zeolite is enhanced. With increasing slag loading, the active material involved in the alkali-excitation reaction increases, the chemical reactivity increases, the content of inert quartz in the mixture decreases, as well as the yield of C-A-S-H gel increases.

3.4. FT-IR Characterization of Brown Coal Gangue-blast Furnace Slag Geopolymer Binder

Figure 6 shows the FT-IR spectra (range 400cm-1 to 4000cm-1) of the samples with 0% and 30% slag addition. The bending vibration of -OH and the stretching vibration absorption peaks of H-O-H in all samples are found at about 3450cm^{-1} and 1650cm^{-1}, indicating the presence of chemically bound water in the samples. The bending vibration of -OH and the stretching vibration absorption peaks of H-O-H for the samples with 0% and 30% slag addition are 3448.28cm^{-1}, 1641.77cm^{-1}, 3448.17cm^{-1} and 1642.27cm^{-1}, respectively. In the Figure, 1429.86cm^{-1} is the peak of C-O stretching vibration band, and no obvious C-O stretching vibration peak was detected for the 0% slag addition sample. In the previous study, the C-O stretching vibration band appeared at the position of about 1440cm^{-1}, and it can be seen that the relative absorption intensity of the peak increased with increasing CaO content.

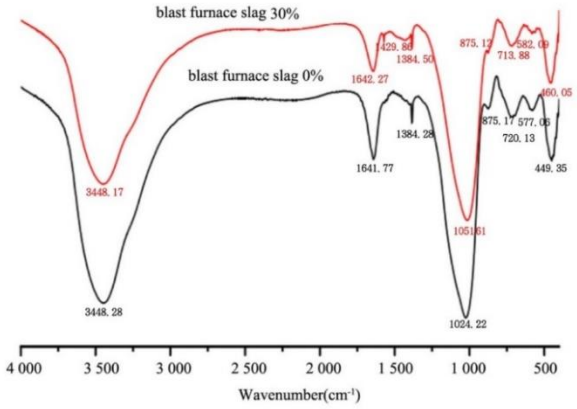


Figure 6. *FT-IR characteristics of geopolymer binder with blast furnace slag dosage.*

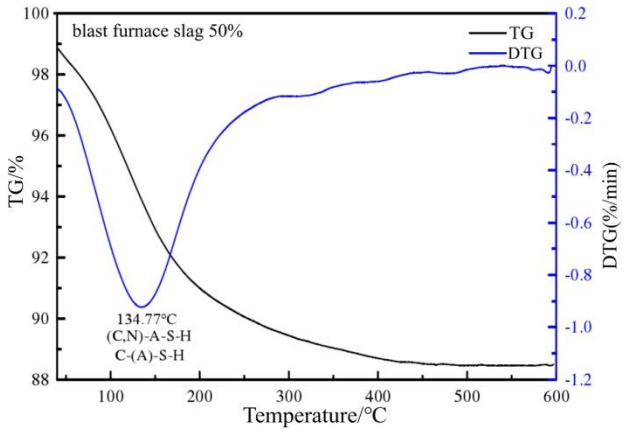
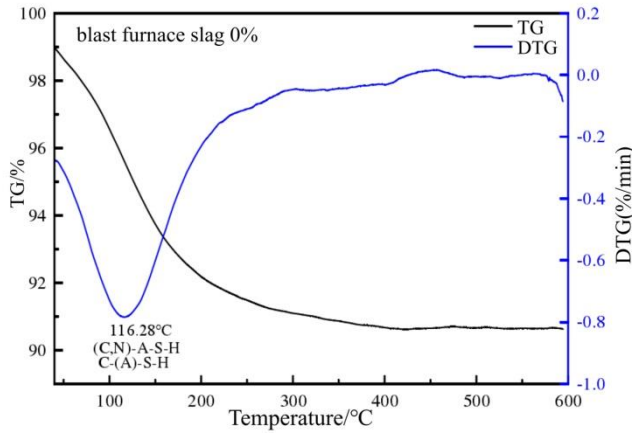
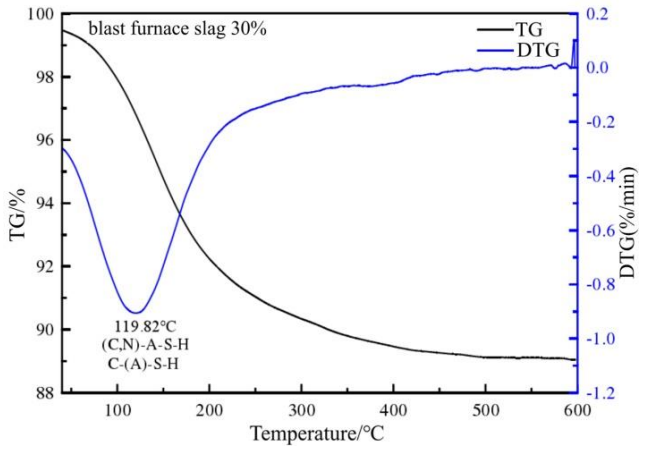


Figure 7. *FT-IR analysis of samples with 0%, 30% and 50% blast furnace slag addition.*

3.5 TG-DTG Analysis of Brown Coal Gangue-blast Furnace Slag Geopolymer

Figure 7 shows the TG-DTG curves of the samples (40℃ to 600℃) with a fixed molar concentration of NaOH of 10M and varying the blast furnace slag dosage of 0%, 30% and 50%.





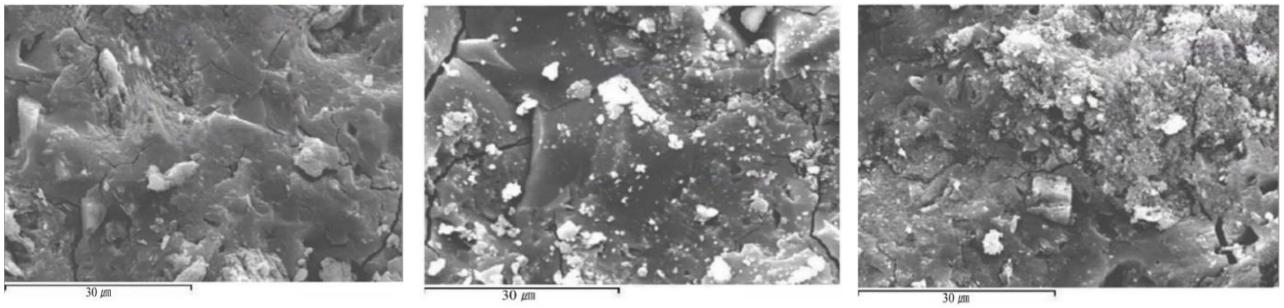
The weight loss peak of the samples is generally around 120℃, and the TG-DTG test results show that the maximum weight loss temperature of the samples with 0%, 30% and 50% blast furnace slag addition is 116.28, 119.82, and 134.77 ℃, respectively. That is, the weight loss peak temperature of the sample shows an increase with increasing blast furnace slag dosage. Before 250℃, the weight loss of aluminosilicate hydrates corresponds to 7.50%, 8.43% and 8.83%, respectively. With the increase of blast furnace slag dosage, the content of C-(A)-S-H increases with the substitution of Na with Ca. With the increase of the yield of hydroalumina silicate, the structure becomes more compact and the compressive strength increases, which is consistent with the compressive strength measurements. Then, the total weight loss of the samples with 0%, 30% and 50% slag addition are 8.39%, 9.83% and 10.98%, respectively.

3.6. SEM Analysis of Alkali Activated Brown Coal Gangue-blast Furnace Slag Geopolymer

To better understand the influence of blast furnace slag through SEM images, we selected only 0%, 30% and 50% slag samples and analyzed them, and the results are shown in Figure 8. It is clearly seen that the microstructural difference between the three samples is significant. The structure of brown coal gangue geopolymer samples with 0% slag loading is relatively poor in surface state and there is clearly very little gel product, i.e. flocculent product. It can be observed that with increasing slag dosage, the bulk and flocculent crystals on the surface increase significantly, and there are also some spongy calcium aluminum hydrate particles on the surface. The main polymerization products of the alkali activated brown coal gangue geopolymer samples are N-A-S-H gel and amorphous aluminosilicate gel, while the main polymerization products of the alkali activated brown coal gangue-blast furnace slag geopolymer samples are N-A-S-H gel, C-(A)-S-H gel and amorphous aluminosilicate gel. There are also some spongy

calcium-aluminum hydrate particles on the surface. In the case of the addition of blast furnace slag to brown coal gangue, the polymer flocculation matrix becomes denser.

Under alkaline conditions, the brown coal gangue and blast furnace slag are well combined, which increases the compressive strength of the samples.



a) blast furnace slag 0% b) blast furnace slag 30% c) blast furnace slag 50%

Figure 8. *SEM of geopolymer binder versus blast furnace slag content.*

Also, there is a uniform microcrack in the sample, which is mainly caused by the drying shrinkage of the alkali-activated sample. With increasing slag loading, C-(A)-S-H gels with high Ca/Si, Ca/Al and Si/Al ratios were produced. Moreover, with the increase of slag content, the Si-Al ratio of brown coal gangue-blast furnace slag samples increases. The Si-Al in coal waste and slag participates in the whole alkali-activated reaction process, with the increase of slag addition, the Si-Al ratio increases and correspondingly, the compressive strength increases. In other words, the increase in blast furnace slag content leads to the formation of C-(A)-S-H gel with relatively high Ca/Si, Ca/Al and Si/Al ratios, which can be attributed to the large increase in the compactness and compressive strength of the samples.

3.7. Effect of Geopolymer Binder and Silica Fume Addition on the Mechanical Strength of Briquettes

Table 2 shows the mechanical strength of geopolymers with the change of alkaline activation solution. From Table 2, the ratio of silica fume/NaOH has some effect on the mechanical strength of the briquettes. When the silica fume/NaOH ratio is 3, the Compressive strength and the strength after heat treatment are 6.9MPa and 4.5 MPa, respectively.

On the other hand, at different silica fume/NaOH ratios, the highest mechanical strength is obtained when the specific gravity of the activation solution is 1.42, while at silica fume/NaOH ratio is 3 and the specific gravity is 1.46, the mechanical strength is rather low. This may be attributed to the increased viscosity of the activation solution at too high specific gravity, which is not sufficient for the dissolution of Si-O tetrahedra and Al-O tetrahedra into the activation solution, and the poor geopolymerization. On the other hand, the mechanical strength of the is low at the silica fume/NaOH ratio of 3.2. This may be due to the fact that a large amount of anhydrous silicic acid dissolves in alkaline solution, the density of the solution increases rapidly, reaches saturation, and the viscosity increases rapidly, leading to gelation. It can be seen that the conditions for the preparation of a suitable activation solution that can be used for the preparation of geopolymer with rapid preparation and controlled reaction rate are silica fume/NaOH ratio of 3 and specific gravity of 1.42.

Figure 9 shows the measured cold strength and post-heat treatment strength of anthracite briquette with varying geopolymer binder dosage of 2-10%. As shown in the Figure, the trend of the increase in cold strength and post-heat treatment strength was observed with the increase in the geopolymer binder dosage. The economic aspects and characteristics of the briquette binder are summarized in Table 1, which shows that the optimum binder dosage is 6%.

Table 2. *Mechanical strength of briquette with varying NaOH molar concentration and specific gravity in alkaline activated solution.*

No	silica fume/NaOH	specific gravity	compressive strength/MPa	compressive strength after heat treatment/MPa
1	1.5	1.33	3.50	2.6
2	2	1.38	4.5	3.2
3	2.5	1.4	6.3	4.2
4	3	1.42	6.9	4.5

No	silica fume/NaOH	specific gravity	compressive strength/MPa	compressive strength after heat treatment/MPa
5	3.2	1.46	6.7	4.2

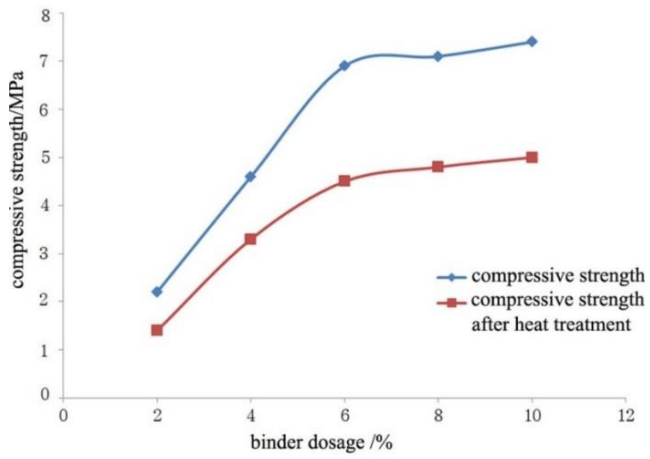


Figure 9. Mechanical strength of briquette with binder dosage.

4. Conclusions

Briquette binder from industrial waste geopolymer was prepared and characterized. The brown coal gangue roasted at 700 ℃ was more activated than the original state. The optimum silica fume/NaOH ratio in the activated solution is 3, specific gravity is 1.42, and molar concentration of NaOH is 10M. The addition of 30% blast furnace slag increases the mechanical strength of geopolymer binder. The optimum dosage of geopolymer binder is 6%, and the compressive strength and post-heat treatment strength of the pellets are 6.9MPa and 4.5MPa, respectively.

Author Contributions

Jin Hyok Ri: Project administration, Investigation
Gwang Song Yun: Formal Analysis
Yong Bom Hong: Methodology
Tok Hui Ri: Software
Myong Il Kim: Writing – review & editing
Hyen Chol Sim: Writing – review & editing
Su Chol Zhang: Validation

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Conflicts of Interest

The authors declare no conflicts of interest.

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